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Wilkinson, R

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CURING RESIN INFUSED COMPOSITES IN THE AUTOCLAVE

Robert Wilkinson, Richard K. Cullen and John Summerscales*

School of Engineering, Plymouth University, Plymouth PL4 8AA, United Kingdom,
* jsummerscales@plymouth.ac.uk, https://www.plymouth.ac.uk/acmc

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ABSTRACT

There is increasing interest across the range of composites manufacturing processes for cost reduction with a current focus on out-of-autoclave (OOA) processes. However, for the highest performance composites, the maximum fibre volume fraction is limited by the compressibility characteristics of the reinforcement. For any specific reinforcement, vacuum-only processes cannot achieve fibre contents as high when additional external pressure is applied. Compression moulding in a hydraulic press creates limited compaction perpendicular to the line of action of the press. The autoclave is good for complex three-dimensional components. Autoclave processes normally use pre-impregnated reinforcements with a premium price for the impregnation process and the associated quality issues. The use of dry reinforcements infused with liquid resins should lead to significant cost reductions. This paper considers the optimisation of autoclave cure for resin-infused composites and extends an earlier feasibility study for composite plates referenced to equivalent systems manufactured by hand-lamination, or by resin infusion without autoclave cure. Consolidation at 5.9 bar lead to an additional 8.4% (thickness method) or 8.6% (burnoff) fibre volume fraction. In turn, the flexural modulus was increased by 39% and the flexural strength was increased by 20% relative to vacuum-only cured composites.

1 INTRODUCTION

The composites industry is especially relevant to transport industries where reduced vehicle mass can improve the energy efficiency and contribute to either reduced fuel consumption or enhanced performance. For cost and process time reasons there is increasing interest in out-of-autoclave processes, e.g. vacuum-bag only (VBO) prepreg, or resin infusion under flexible tooling (RIFT) [1-3]. However, it is inherent in the compressibility characteristics of any specific reinforcement fabric [4-6] that limiting the pressure during manufacture (to one atmosphere) will compromise the maximum achievable fibre volume fraction (FVF). Reduced FVF implies parasitic resin mass due to the increased matrix volume fraction. The consequent resin-rich volumes (RRV), where larger voids can occur, act as stress concentrations with negative effects on material strengths. Textile reinforcements with clustered fibres generally have low strength but high in-plane permeability, whereas the opposite is true for uniformly distributed fibres [7].

This paper considers the optimisation of autoclave cure for resin-infused composites and extends an earlier feasibility study [8] for composite plates referenced to equivalent systems manufactured by hand-lamination, or by resin infusion without autoclave cure.

2 MATERIALS AND METHODS

All experiments were conducted using a single roll of 270 gm^{-2} plain-woven biaxial glass fibre fabric with the warp fibres parallel in all plies. The matrix was EasyComposites IP2 unsaturated polyester resin with Butanox M50 MEKP catalyst. All laminates were manufactured with 10 plies of reinforcement using resin infusion under flexible tooling with a flow medium (RIFT II). Laminate preparation prior to loading the autoclave was conducted under ambient conditions.

To investigate the effect of resin dwell times, viscosity tests were conducted using a Brookfield R/S CPS-P rheometer with a C50-1 cone and a 20°C plate. The thin film test was assumed to provide similar viscosities during cure to those for resin confined by the reinforcements. Variable rate shear

tests were used to determine that the resin was a Newtonian fluid. At 2% catalyst, the time from an initial mix viscosity of 250 mPa.s to specific viscosities was established to be 39.5 min to 500 mPa.s, 45 min to 1000 mPa.s and 48.3 min to 2000 mPa.s.

Initial experiments were conducted with a (dry) fabric reservoir inside the vacuum bag downstream of the wetted infused laminate. When the autoclave pressure was applied with both inlet and outlet pipes clamped, there was no significant change in FVF relative to that for the plate cured at ambient pressure.

Subsequent tests used no reservoir material, and the resin inlet was clamped while the resin outlet was vented to atmosphere during autoclave consolidation. The outlet pipe used 6 m of 6 mm ID pipe coiled around a cylinder to capture the resin expelled from the laminate. After infusion, plates were subjected to (a) vacuum-bag only pressure, (b) 3.1 bar pressure in the autoclave or (c) 5.9 bar pressure in the autoclave. Further laminates were prepared and pressurised after a dwell period to study the effect of viscosity at the time pressure was applied for the four times identified by the viscosity tests.

All laminates were post-cured at 80°C for 5 h. FVF was determined from both panel thickness (CRAG method 1000) and by weighing in both air and water. Elastic moduli for the laminates were estimated using the rule-of-mixtures (RoM) and simulated in Autodesk Helius Composite 2016 laminate analysis (LA) software.

Samples from the laminates were tested to determine the flexural modulus and strength (σ'_f : BS EN ISO 14125:1998+A1:2011) and interlaminar shear strength (ILSS: BS EN ISO 14130:1998) using an Instron 5582 test frame with a 100 kN load cell. Flexure tests were conducted at 1 mm/min, while ILSS tests were conducted at 2 mm/min.

Specimens for optical microscopy were cut to expose either the warp, or weft, direction fibres. They were potted in polyester resin then polished on a Buehler Automet 250. Optical microscopy used an Olympus SC50 optical microscope with Olympus Stream software. No gross voids were detected and potential small voids may have been polishing artefacts. Further examination of microstructures and fracture surfaces was undertaken with a JSM-6610LV scanning electron microscope and SkyScan 1174 micro-computed tomography x-ray scanner.

3 RESULTS

All manufacturing experiments are based on single runs, while mechanical testing used at least five specimens with valid failure modes. Table 1 summarises data acquired from the panels manufactured with the outlet pipe vented to air.

Experiment		Infusion	3.1/0.0	5.9/0.0	5.9/39.5	5.9/45	5.9/48.3
External pressure	[bar]	0.0	3.1	5.9	5.9	5.9	5.9
Dwell time	[min]	0.0	0.0	0.0	39.5	45.0	48.3
Thickness	[μm]	2040±51	1890±3	1780±16	1880±1	1930±11	1980±1
FVF (thickness)	[%]	51.9±1	56.1±0.1	59.3±0.5	56.3±0.0	54.7±0.3	53.6±0.0
FVF (burn-off)	[%]	52.3	56.4	60.9	56.7	54.3	52.4
Modulus (expt)	[GPa]	20.5±0.4	25.6±0.9	28.4±0.9	26.4±0.7	25.4±0.5	24.0±1.0
Modulus (RoM)	[GPa]	21.9	23.4	24.9	23.5	22.6	22.0
Modulus (LA)	[GPa]	22.4	24.2	26.5	24.4	23.3	22.4
Initial σ'_f	[MPa]	321±15	338±23	375±17	324±17	284±24	325±21
Ultimate σ'_f	[MPa]	347±14	384±16	415±20	391±14	383±10	364±12
Initial ILSS	[MPa]	35.6±1.6	39.6±1.8	37.1±2.8	36.6±4	40.0±1.6	35.9±1.8
Ultimate ILSS	[MPa]	41.0±1.9	44.5±2.5	41.5±2.5	42.5±3.4	43.2±1.8	42.4±1.7

Table 1: Summary data for panels manufactured with the outlet pipe vented to air.

Fig. 1 presents laminate fibre volume fractions against applied external pressure/dwell time. Increasing consolidation pressure with pressure applied immediately after infusion resulted in increased FVF. Under similar conditions (albeit reservoir material previously and pipe here), Lewin et al [8] reported only 0.26% increase in FVF when the panel was not vented to atmosphere, whereas the

equivalent panel in this study had an 8.6% increase in FVF. At constant consolidation pressure, delaying the consolidation resulted in reduced FVF.

Fig. 2 shows flexural moduli versus FVF from burn-off. The y-axis intercept has been forced to the modulus of the resin. RoM and LA predictions validate the trend with R^2 values of 1 and 0.945 respectively.

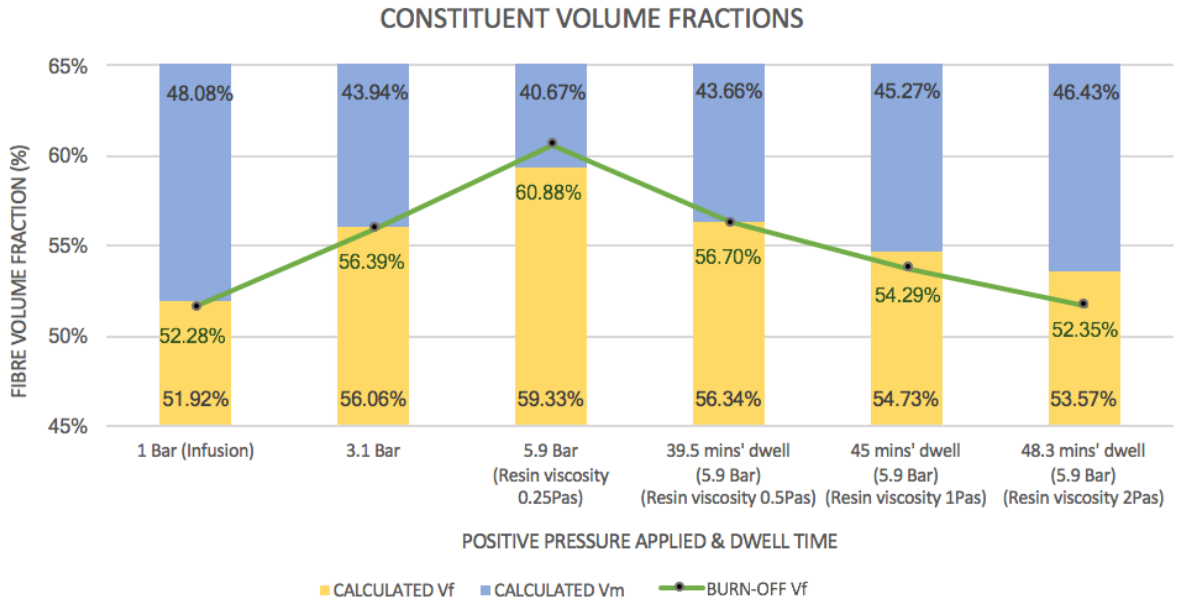

Figure 1: Laminate fibre volume fractions versus against applied external pressure/dwell time.

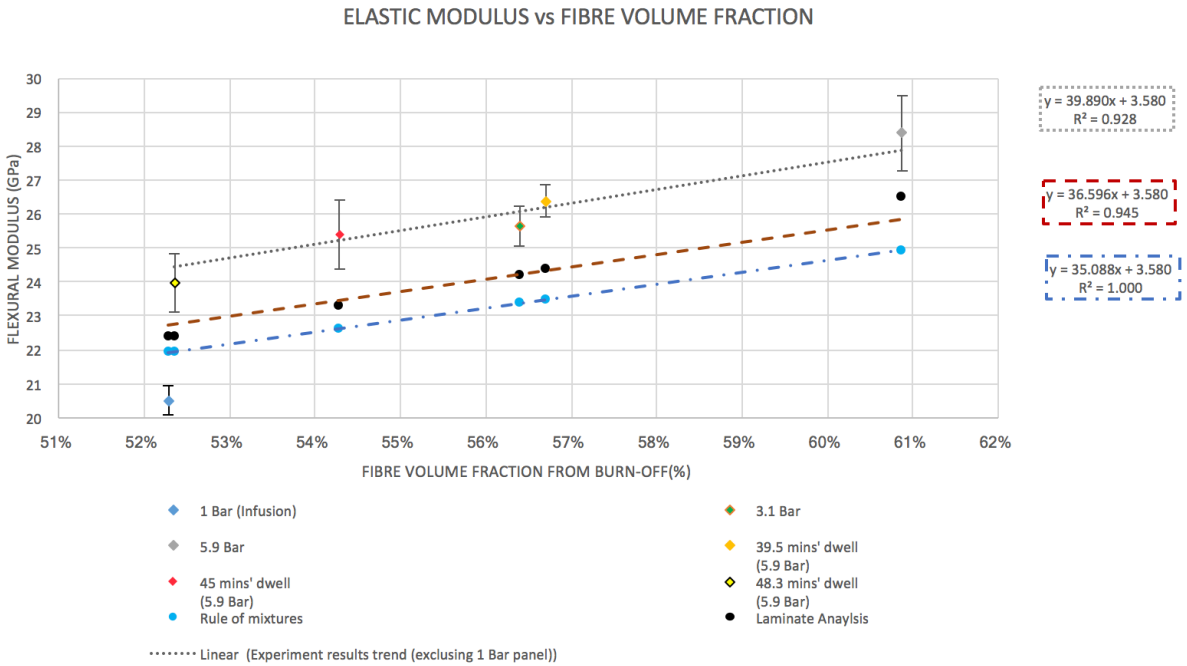

Figure 2: Flexural moduli versus FVF from burn-off

Fig. 3 presents flexural strength versus applied positive pressure during consolidation. Flexural strength increased with increasing consolidation pressure. Initial strengths are based on the first load-drop, whereas ultimate strengths are based on peak load. Fig. 4: shows flexural strength versus viscosity at time of application of autoclave pressure. Increased viscosity limited the quantity of resin expelled from the laminate, reduced the FVF, and resulted in lower mechanical properties.

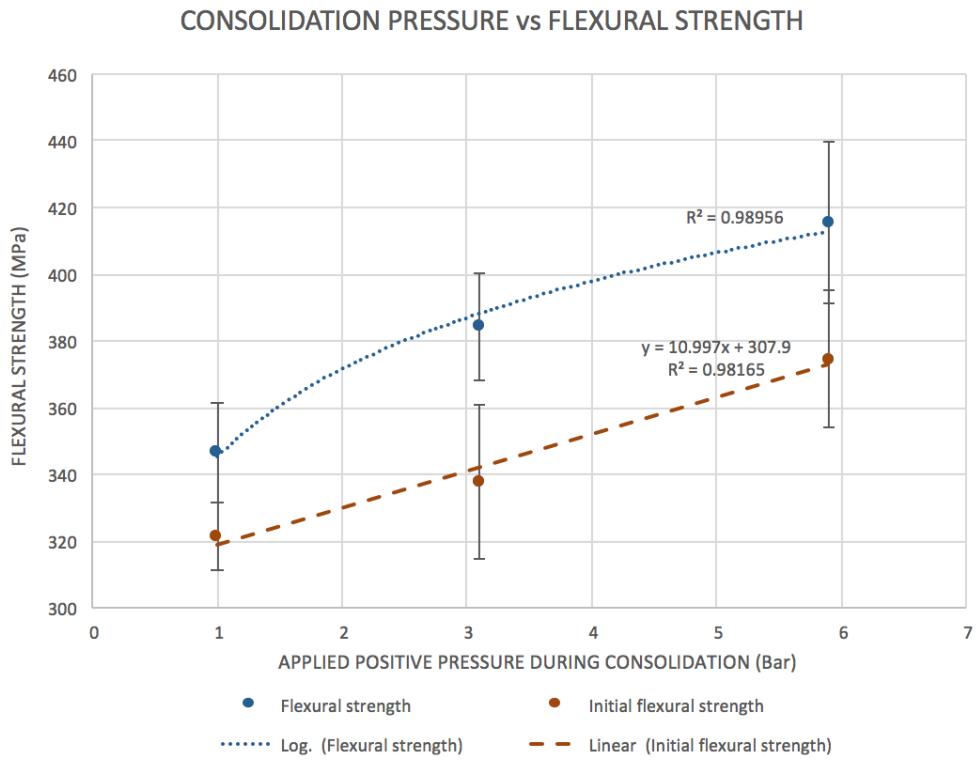

Figure 3: Flexural strength versus applied positive pressure during consolidation.

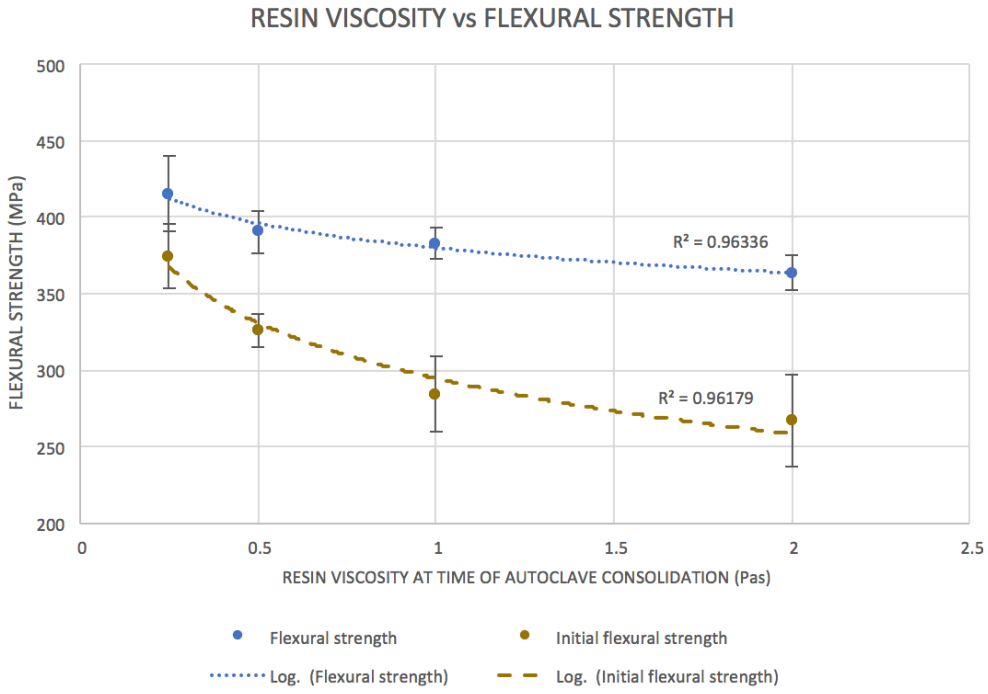

Figure 4: Flexural strength versus viscosity at time of application of autoclave pressure.

Fig. 5 and Fig. 6 show Inter-Laminar Shear Strength (ILSS) against manufacturing conditions or against FVF from burn-off respectively. The ILSS obtained indicate that the fibres are well-bonded to the matrix, with no significant difference between the comparable values between different process conditions.

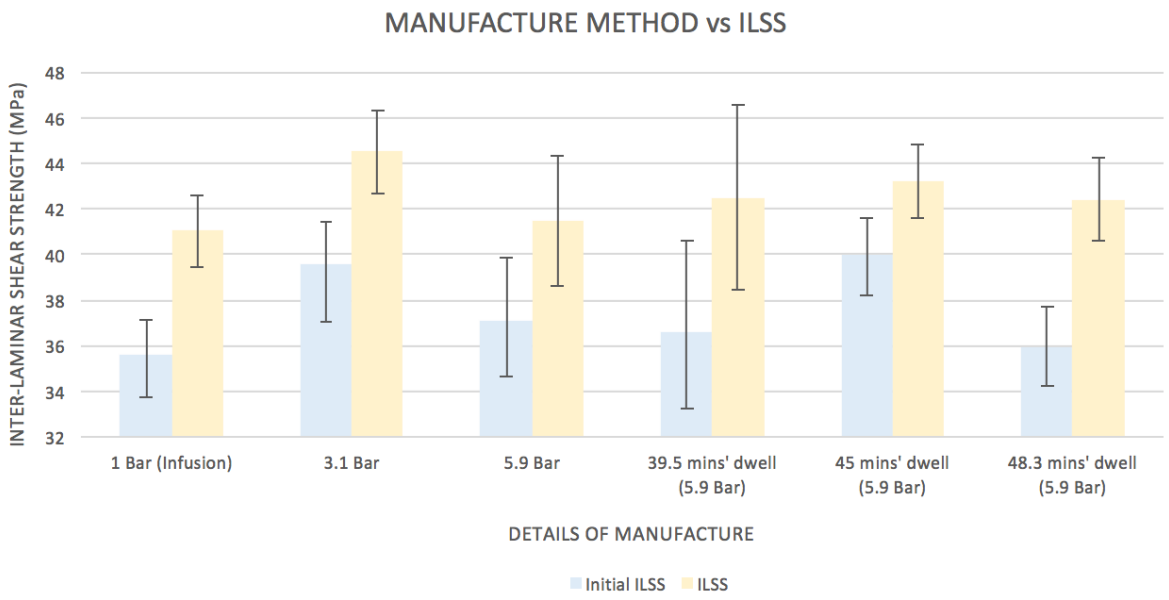

Figure 5: Inter-Laminar Shear Strength (ILSS) versus manufacturing conditions.

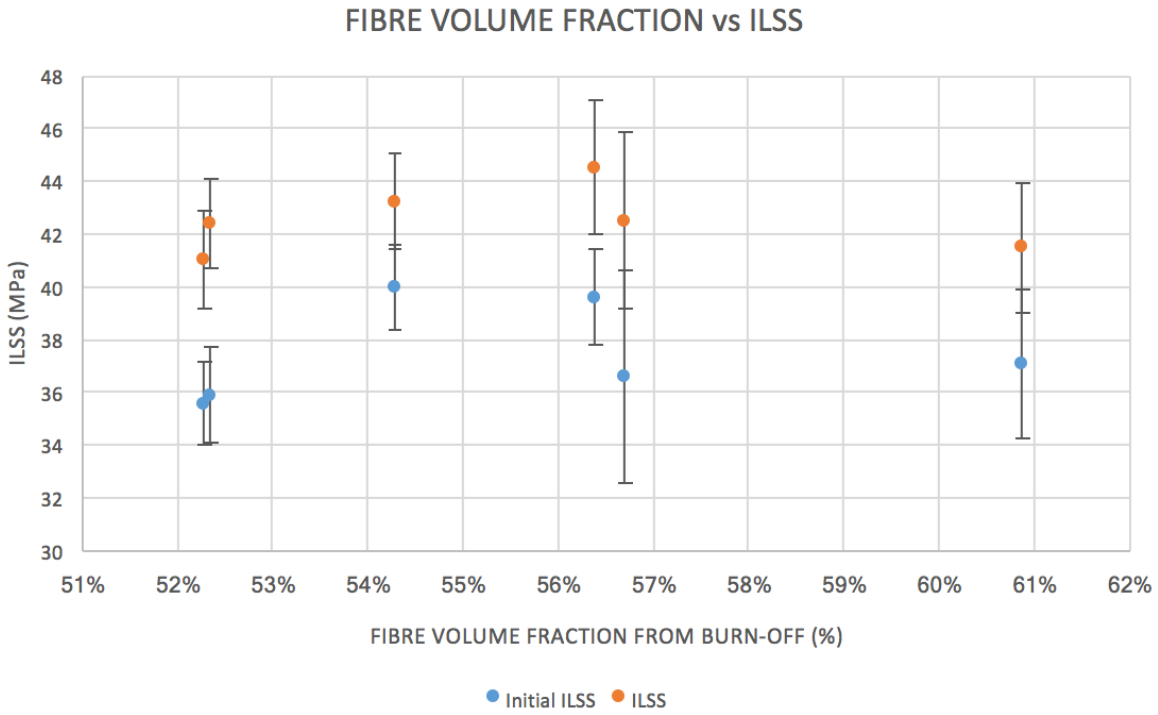

Figure 6: Inter-Laminar Shear Strength (ILSS) versus FVF from burn-off.

Fig. 7 and Fig. 8 show the microstructures of the composites produced permitting qualitative comparisons of the extent of resin-rich volumes. Resin-rich volumes (observed as resin-rich areas in polished microstructures images) decrease with increasing FVF. A small number of microvoids were detected in low-pressure consolidated laminates.

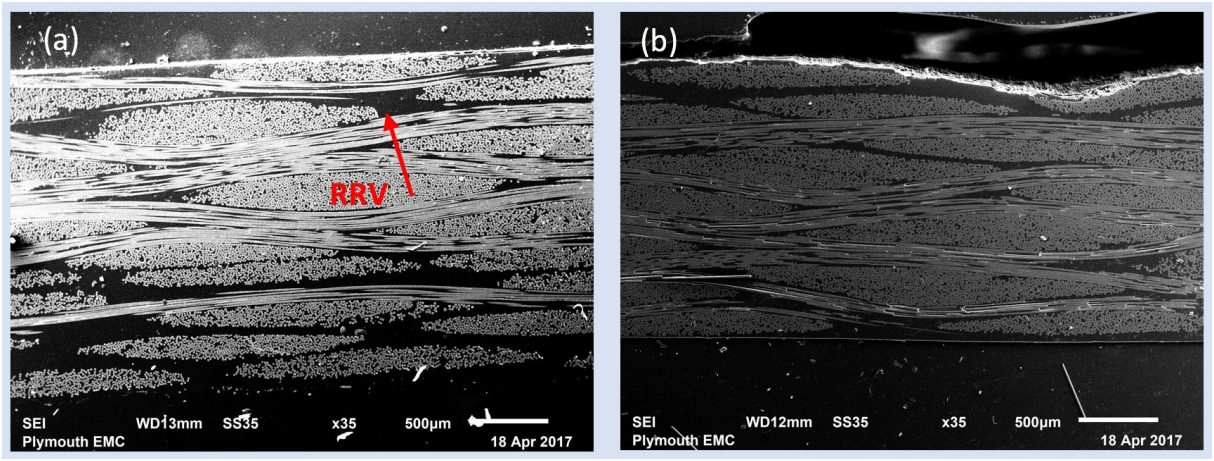

Figure 7: SEM images showing (a) resin-rich volumes (RRV) in infusion only laminate, and (b) reduced levels if RRV in the 5.9 bar consolidated laminate.

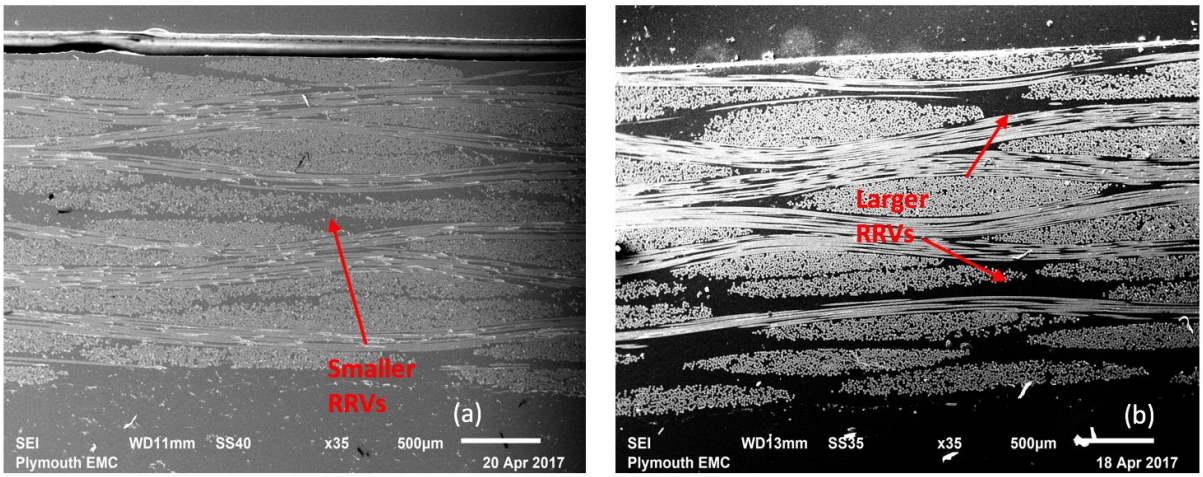

Figure 8: SEM images comparing resin-rich volumes (RRV) in (a) 5.9 bar 48 min dwell consolidated laminate, and (b) infusion only laminate.

4 DISCUSSION

The data acquired in this study suggests that the highest FVF laminates result when consolidation in the autoclave immediately follows infusion. The low resin viscosity during consolidation permits better removal of excess resin from the laminate in the in-plane flow situation.

For laminates cured with an extended dwell time, the increase in resin viscosity limited any increase in FVF. The low volume fraction for composites consolidated at 2000 mPa.s is consistent with the expectation that the in-plane flow front is effectively stationary at this viscosity and the low pressures used in liquid composite moulding processes. Becker [9] quotes an upper limit for viscosity in resin transfer moulding (RTM) of 800 mPa.s, while elsewhere the non-injection point (NIP) is defined as a viscosity of 1000 mPa.s [10].

It is interesting to note that Stringer [11] identified that, for the through-thickness flow in wet lay-up/vacuum bag process for carbon fibre/epoxy resin composites, the viscosity at the start of the consolidation was the critical parameter in the achievement of high fibre volume fraction and low void content. A dwell time window was identified which "exists between the same viscosity limits regardless of the resin system and temperature being used". CFRP composites with FVF up to 58% and void volume fraction below 2% were obtained with a dwell time window of 7500-16500 mPas (75-165 poise).

Given that:

a) prepreg is typically three-times the cost of dry fabric,
b) that the breather and bleeder fabric used for prepreg are replaced by flow medium in infusion, and
c) the cost of nylon pipe is similar to the cost of one square metre of dry fabric (and the required length could be reduced in an optimised process),

the materials costs should be significantly lower for the infusion/autoclave process relative to the prepreg/autoclave process. Further, the laminate can be loaded to the autoclave on completion of infusion without a dwell time, so autoclave cycles will be shorter allowing more components to be processed in any given period.

5 CONCLUSIONS

The limited set of experiments undertaken have demonstrated a novel method for autoclave consolidation of resin-infused composites. Plates infused, then immediately loaded and pressurised in the autoclave, had higher fibre volume fractions and a noticeable (quantitative assessment) reduction in resin-rich volumes. This lead to increased mechanical properties relative to either unconsolidated laminates or laminates where consolidation took place at the same pressure after a short dwell time.

The 5.9 bar consolidation pressure lead to an additional 8.4% (thickness method) or 8.6% (burn-off) fibre volume fraction. In turn, the flexural modulus was increased by 39% and the flexural strength was increased by 20% relative to vacuum-only cured composites. The flexural strength decreased with increasing dwell time /increasing viscosity.

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